## B.7.6 Residues Resulting from Supervised Trials (Annex IIA 6.3; Annex IIIA 8.3)

### B.7.6.1 Residues in Target Crops

### B.7.6.1.1 Crop Subgroup 13-07B

| | |
|---|---|
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| **Guidelines:** | EPA OCSPP Harmonized Test Guideline 860.1500 Crop Field Trials (August 1996)<br>PMRA Regulatory Directive DIR98-02 – Residue Chemistry Guidelines, Section 9 – Crop Field Trials<br>PMRA Regulatory Directive DIR2010-05 – Revisions to the Residue Chemistry Crop Field Trial Requirements<br>OECD Guideline 509 Crop Field Trial (September 2009) |
| **GLP Compliance:** | No deviations from regulatory requirements were reported which would have an impact on the validity of the study. |
| **Acceptability:** | The study is considered scientifically acceptable. |
| **Evaluator:** | Jack Giordano, Chemist, RAB2/HED (7509P) |

## EXECUTIVE SUMMARY

Twelve field trials for sulfoxaflor on blueberries were conducted in Canada and the United States (US) encompassing North American Free Trade Agreement (NAFTA) Growing Regions 1 (1 trial in Maine), 1A (3 trials in Nova Scotia), 2 (3 trials in Georgia and New Jersey), 5/5A/5B (3 trials in Michigan), and 12 (2 trials in British Columbia and Oregon) during the 2014 growing season. All trials met the "Criteria for Determining Independence of Crop Field Trials" (November 2014) and are considered independent.

At each trial location, sulfoxaflor, formulated as a suspension concentrate (Closer SC, EPA Reg. 62719-623 and PCP # 30826), was applied to blueberries as three foliar directed or broadcast applications at rates of 0.089-0.094 lbs a.i./A (98.6-105.7 g a.i./ha). The re-treatment intervals were 6-8 days, and the total application rates were 0.267-0.279 lbs a.i./A (298.9-313.1 g a.i./ha). An adjuvant was added to the spray mixture for all applications. Blueberries were harvested at a preharvest interval (PHI) of 1 day. In two trials, samples were collected at different time intervals (PHIs of 0, 8, 14, and 21 days at trial site NS321, and PHIs of 0, 7, and 14 days at trial site OR399) to monitor residue decline.

All samples were maintained frozen at the testing facility, during shipping to the laboratory, and were stored frozen until analysis. The maximum storage interval for samples between harvest and extraction was 723 days (~24 months). Residues of sulfoxaflor have been shown to be stable in blueberries for up to 756 days (~25 months) under frozen conditions. Adequate storage

stability data are therefore available to support the storage conditions and intervals for samples in the current trials.

Samples in the current study were analyzed using Method 091116, a LC/MS/MS method to determine residues of sulfoxaflor and metabolites X11719474 and X11721061. Acceptable method validation and concurrent recoveries were reported for blueberry samples at fortification levels of 0.01-2.0 mg/kg (ppm), thus validating the method. The limit of quantitation (LOQ), based on the lowest level of method validation, was 0.01 ppm per analyte for blueberries.

Individual sample (and per-trial average) residues in blueberries ranged from <0.146 ppm to <1.419 ppm (<0.175 ppm to <1.293 ppm). Residue decline data show that combined residues of sulfoxaflor, X11719474, and X11721061 decrease in blueberries with increasing PHIs.

## I. MATERIALS AND METHODS

## A. MATERIALS

| **Table B.7.6.1.1-1. Nomenclature for Sulfoxaflor and Metabolites of Interest.** | |
|---|---|
| **Common name** | Sulfoxaflor |
| **Identity** | *N*-[methyloxido[1-[6-(trifluoromethyl)-3-pyridinyl]ethyl]-$\lambda^4$-sulfanylidene]cyanamide |
| **CAS no.** | 946578-00-3 |
| **Company experimental name** | XDE-208 (Dow Agro)<br>ASF 1069 (Syngenta) |
| | |
| **Metabolite** | X11719474 |
| **Identity** | *N*-((methyl)oxido)(1-[6-(trifluoromethyl)pyridine-3-yl]ethyl)-$\lambda^4$-sulfanylidene)urea |
| | |
| **Metabolite** | X11721061 |
| **Identity** | 1-[6-(trifluoromethyl)pyridine-3-yl]ethanol |

## B. Study Design

## 1. Test Procedure

A total of 12 residue trials in/on blueberries were conducted with a suspension concentrate of sulfoxaflor (Closer SC) during the 2014 growing season (Table B.7.6.1.1-2).

| **Table B.7.6.1.1-2. Trial Numbers and Geographical Locations.** | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Crop | Region | | | | | | | | | | | | | | Total |
| | 1/1A | 2 | 3 | 4 | 5/5A/5B | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | |
| Blueberries | 4 | 3 | -- | -- | 3 | -- | -- | -- | -- | -- | -- | 2 | -- | -- | 12 |

| Table B.7.6.1.1-2B. Independent Trial Determination[1] | | | |
|---|---|---|---|
| Crop | Trial Nos. | Differences | Decision |
| Blueberries | MI276, MI277, and MI278 | Independently prepared tank mixes at each site: The tank mix composition used at each site is different. As such independently prepared tank mixes were used at each site.<br><br>Location: All trials were conducted in Fennville, MI.<br><br>Timing: The applications at each trial site were made less than 30 days apart from each other.<br><br>Variety: Trial MI276 used Rubel blueberries, MI277 used Bluecrop blueberries, and MI278 used Jersey blueberries. | Separate due to independently prepared tank mixes and different varieties. |

[1] All assessments are based on the replicate trial guidance presented in draft memo 568_Criteria for Independence of Trials 04/23/2013 (EPA) and final memo Criteria for Independence of Crop Field Trials November 2014 (PMRA).

Locations and detailed use patterns for the trials are provided in Table B.7.6.1.1-3.

| Table B.7.6.1.1-3. Study Use Pattern. | | | | | | | |
|---|---|---|---|---|---|---|---|
| Location: City, State/Province; Year (Trial ID)[1] | End-use Product/ Formulation (% a.i.) | Method of Application/ Timing of Application | Volume (gal/A) [L/ha] | Rate per Application (lbs a.i./A) [g a.i./ha] | Retreatment Interval (days) | Total Rate (lbs a.i./A) [g a.i./ha] | Surfactant/ Adjuvant |
| Langley, BC; 2014 (BC23) | Closer SC/ Suspension concentrate (2 lbs a.i./gal) [240 g a.i./L] | 1. Foliar directed/ Fruiting | 54 [503] | 0.092 [102.8] | -- | 0.275 [307.6] | Agral 90 NIS |
| | | 2. Foliar directed/ Fruiting | 53 [498] | 0.091 [101.8] | 7 | | |
| | | 3. Foliar directed/ Fruiting | 54 [505] | 0.092 [103.0] | 7 | | |
| Alapha, GA; 2014 (GA*187) | Closer SC/ Suspension concentrate (2 lbs a.i./gal) [240 g a.i./L] | 1. Foliar directed/ Fruiting | 29 [274] | 0.088 [98.6] | -- | 0.267 [298.9] | UCPA Surfactant 80/20 NIS |
| | | 2. Foliar directed/ Fruiting 50% | 30 [281] | 0.090 [101.9] | 7 | | |
| | | 3. Foliar directed/ Fruiting | 29 [276] | 0.089 [99.2] | 8 | | |
| Jonesboro, ME; 2014 (ME242) | Closer SC/ Suspension concentrate (2 lbs a.i./gal) [240 g a.i./L] | 1. Foliar broadcast/ Fruiting | 25 [238] | 0.091 [102.2] | -- | 0.271 [303.7] | Dyne-Amic vegetable oil and silicone surfactant |
| | | 2. Foliar broadcast/ Fruiting | 25 [235] | 0.090 [100.8] | 8 | | |
| | | 3. Foliar broadcast/ Fruiting | 25 [235] | 0.090 [100.7] | 6 | | |
| Fennville, MI; 2014 (MI276) | Closer SC/ Suspension concentrate (2 lbs a.i./gal) [240 g a.i./L] | 1. Foliar directed/ Fruiting | 36 [339] | 0.092 [103.2] | -- | 0.279 [313.3] | Activator 90 NIS |
| | | 2. Foliar directed/ Fruiting | 37 [343] | 0.093 [104.4] | 7 | | |
| | | 3. Foliar directed/ Mature fruit | 37 [348] | 0.094 [105.7] | 7 | | |
| Fennville, MI; 2014 (MI277) | Closer SC/ Suspension concentrate (2 lbs a.i./gal) [240 g a.i./L] | 1. Foliar directed/ Fruiting | 74 [692] | 0.090 [101.4] | -- | 0.269 [302.4] | SuperSpread 7000 NIS |
| | | 2. Foliar directed/ Fruiting | 73 [684] | 0.089 [100.3] | 6 | | |
| | | 3. Foliar directed/ Fruiting | 75 [705] | 0.090 [100.7] | 7 | | |
| Fennville, MI; 2014 (MI278) | Closer SC/ Suspension concentrate (2 lbs a.i./gal) [240 g a.i./L] | 1. Foliar directed/ Fruiting | 55 [513] | 0.090 [101.0] | -- | 0.270 [302.4] | SuperSpread 7000 NIS |
| | | 2. Foliar directed/ Fruiting | 55 [518] | 0.090 [100.8] | 7 | | |
| | | 3. Foliar directed/ Fruiting | 56 [527] | 0.090 [100.6] | 7 | | |
| Cream Ridge, NJ; 2014 (NJ306) | Closer SC/ Suspension concentrate (2 lbs a.i./gal) [240 g a.i./L] | 1. Foliar directed/ Green fruit | 30 [283] | 0.091 [101.6] | -- | 0.270 [302.7] | MSO |
| | | 2. Foliar directed/ Green and ripe fruit | 30[283] | 0.090 [101.4] | 7 | | |
| | | 3. Foliar directed/ Fruiting | 29 [275] | 0.089 [99.7] | 7 | | |

**Table B.7.6.1.1-3. Study Use Pattern.**

| Location: City, State/Province; Year (Trial ID)[1] | End-use Product/ Formulation (% a.i.) | Method of Application/ Timing of Application | Volume (gal/A) [L/ha] | Rate per Application (lbs a.i./A) [g a.i./ha] | Retreatment Interval (days) | Total Rate (lbs a.i./A) [g a.i./ha] | Surfactant/ Adjuvant |
|---|---|---|---|---|---|---|---|
| Chatsworth, NJ; 2014 (NJ307) | Closer SC/ Suspension concentrate (2 lbs a.i./gal) [240 g a.i./L] | 1. Foliar directed/ Green fruit | 31 [288] | 0.094 [104.9] | -- | 0.276 [309.7] | Attach NIS |
| | | 2. Foliar directed/ Green fruit | 31 [291] | 0.090 [101.3] | 7 | | |
| | | 3. Foliar directed/ Fruiting | 32 [297] | 0.092 [103.5] | 7 | | |
| Sheffield Mills, NS; 2014 (NS321) | Closer SC/ Suspension concentrate (2 lbs a.i./gal) [240 g a.i./L] | 1. Foliar broadcast/ Fruiting | 38 [356] | 0.092 [103.0] | -- | 0.274 [307.1] | Agral 90 NIS |
| | | 2. Foliar broadcast/ Blue berries present | 38 [351] | 0.090 [101.4] | 7 | | |
| | | 3. Foliar broadcast/ Blue berries present | 38 [356] | 0.092 [102.7] | 7 | | |
| Milford, NS; 2014 (NS322) | Closer SC/ Suspension concentrate (2 lbs a.i./gal) [240 g a.i./L] | 1. Foliar broadcast/ 50% berries blue in colour | 38 [351] | 0.090 [101.2] | -- | 0.270 [302.9] | Agral 90 NIS |
| | | 2. Foliar broadcast/ Blue berries present | 37 [348] | 0.090 [100.4] | 7 | | |
| | | 3. Foliar broadcast/ Mature fruit | 38 [351] | 0.090 [101.3] | 7 | | |
| Debert, NS; 2014 (NS323) | Closer SC/ Suspension concentrate (2 lbs a.i./gal) [240 g a.i./L] | 1. Foliar broadcast/ 90% of berries blue | 38 [351] | 0.090 [101.2] | -- | 0.269 [302.2] | Agral 90 NIS |
| | | 2. Foliar broadcast/ 90% of berries blue | 37 [347] | 0.089 [100.1] | 6 | | |
| | | 3. Foliar broadcast/ Mature fruit | 37 [350] | 0.09 [100.9] | 6 | | |
| Aurora, OR; 2014 (OR399) | Closer SC/ Suspension concentrate (2 lbs a.i./gal) [240 g a.i./L] | 1. Foliar directed/ Fruiting, green and blue | 40 [373] | 0.089 [100.1] | -- | 0.268 [301.0] | Cohere NIS |
| | | 2. Foliar directed/ Fruiting, blue and green | 40 [375] | 0.090 [100.6] | 7 | | |
| | | 3. Foliar directed/ Fruiting, mostly ripe | 40 [374] | 0.089 [100.3] | 7 | | |

[1] All Trial ID #s have the prefix 11296.14-

Blueberries were grown and maintained according to typical agricultural practices. Irrigation was used at trial sites BC23, GA*187, and OR399. Trial sites NJ306 and NJ307 reported rainfall and temperatures to vary around normal, and MI276 had cooler than average temperatures. The rest of the trial sites did not report any unusual weather conditions during the study.

**Sample Handling and Preparation**

Sampling started in the untreated control plot and ended in the treated plot. Berries were handpicked or raked from high/low and exposed/shielded areas on both sides of the row, avoiding row ends. All samples were placed into frozen storage within 3.5 hours of harvest. The samples were shipped frozen to the analytical laboratory (IR-4 Southern Region Laboratory in Gainesville, Florida) by ACDS freezer truck, hand delivery by field personnel, or by FedEx overnight air express packed in dry ice. Two samples from the OR399 trial were mistakenly shipped to the IR-4 North Central Laboratory in Michigan, and were stored there until shipment by ACDS to the analytical laboratory. All samples arrived frozen and intact at the analytical laboratory. The samples were checked in, ground with dry ice and then stored frozen (< -20°C) until extraction and analysis.

**2. Description of Analytical Procedures**

Samples of blueberries were analyzed for residues of sulfoxaflor, X11719474, and X11721061 using a working method very similar to the reference Method 091116, entitled: "Enforcement

Method for the Determination of Sulfoxaflor (XDE-208) and its Main Metabolites in Agricultural Commodities using Offline Solid-Phase Extraction and Liquid Chromatography with Tandem Mass Spectrometry Detection" (PMRA # 1941241). The reference method has been reviewed by PMRA (PMRA # 2313516) and was deemed acceptable for enforcement. Minor modifications were made to the reference method to improve performance, such as centrifuging samples for 5 minutes instead of 3 minutes, making up the mixed internal standard solution in acetonitrile instead of methanol/glycerin, and adding 2.5 ml of sample to the centrifuge tube instead of 500 µl. The sample evaporator was at 60°C instead of 40°C, and the sample was concentrated to about 0.5 ml instead of to near dryness. A Supelclean ENVI-Carb SPE tube was used instead of an Oasis HLB 96 well-plate. The sample was concentrated to reduce the amount of acetonitrile and brought up to the 5 ml mark with 5/95 acetonitrile/water and 0.1% formic acid. The extract was filtered prior to analysis, and different LC/MS/MS model, HPLC column, mobile phases, gradient, and injection volume were used. Finally, negative ionization was used for sulfoxaflor and its internal standard X11843864 instead of positive ionization, as negative ionization provides increased sensitivity and lower background.

Briefly, samples were extracted by homogenizing and shaking with 80/20 acetonitrile/water. Extracts were centrifuged, and then an aliquot of the extract was combined with internal standard solution and evaporated. The extracts requiring dilution were diluted before the addition of internal standard. Aqueous sodium hydroxide was added and hydrolyzed at 50°C. The extracts were acidified with aqueous formic acid and incubated at 50°C with glucosidase from Aspergillus Niger solution. The solution was purified with a Supelclean ENVI-Carb SPE cartridge. Extracts were filtered before analysis using LC/LS/MS with heated electro-spray ionization. The LOQ was 0.01 ppm for each analyte, based on the lowest level of method validation.

## II. RESULTS AND DISCUSSION

Method performance was evaluated during method validation and by use of concurrent recovery by fortifying control samples with 0.01 ppm (n=12), 0.1 ppm (n=5), and 1.0 ppm (n=4) of each analyte. The method was verified at the 2.0 ppm fortification level for sulfoxaflor (n=3). All recoveries were within the acceptable range of 70% to 120% (Table B.7.6.1.1-4); therefore, the method was considered valid for the analysis of residues of sulfoxaflor, X11719474, and X11721061 in blueberry matrices. The fortification levels did bracket the measured residues.

**Table B.7.6.1.1-4. Summary of Method Validation and Concurrent Recoveries of Sulfoxaflor from Blueberries.**

| Matrix | Fortification Level (ppm) | Recoveries (%) | Mean ± Std. Dev. (%) |
|---|---|---|---|
| Sulfoxaflor | | | |
| Blueberries (Method validation) | 0.01 | 104, 107, 107 | 106 ± 1.7 |
| | 0.1 | 82, 83, 84 | 83 ± 1 |
| | 1.0 | 84, 83, 82 | 83 ± 1 |
| | 2.0 | 102, 102, 102 | 102 ± 0 |
| Blueberries (Concurrent recovery) | 0.01 | 108, 109, 105, 114, 103, 113, 105, 104, 105 | 107 ± 4.0 |
| | 0.1 | 85, 82 | 84 ± 2.1 |
| | 1.0 | 82 | -- |
| Metabolite X11719474 | | | |
| Blueberries (Method validation) | 0.01 | 96, 97, 99 | 97± 1.5 |
| | 0.1 | 83, 85, 86 | 85 ± 1.5 |

| Table B.7.6.1.1-4. Summary of Method Validation and Concurrent Recoveries of Sulfoxaflor from Blueberries. | | | |
|---|---|---|---|
| Matrix | Fortification Level (ppm) | Recoveries (%) | Mean ± Std. Dev. (%) |
| | 1.0 | 84, 83, 83 | 83 ± 0.6 |
| Blueberries (Concurrent recovery) | 0.01 | 97, 99, 94, 100, 101, 89, 90, 103, 103 | 97± 5.3 |
| | 0.1 | 85, 82 | 84 ± 2.1 |
| | 1.0 | 81 | -- |
| Metabolite X11721061 | | | |
| Blueberries (Method validation) | 0.01 | 100, 100, 102 | 101 ± 1.2 |
| | 0.1 | 83, 85, 85 | 84 ± 1.2 |
| | 1.0 | 85, 84, 84 | 84 ± 0.6 |
| Blueberries (Concurrent recovery) | 0.01 | 100, 98, 95, 99, 95, 89, 97, 99, 98 | 97 ± 3.4 |
| | 0.1 | 82, 82 | 82 ± n/a |
| | 1.0 | 83 | -- |

The detector response was linear (coefficient of determination, $r^2$ >0.99) within the range of 0.05 ng/ml to 5.0 ng/ml. Representative chromatograms of control samples, fortified samples and treated samples were provided. The control chromatograms generally had no peaks of interest above the chromatographic background. The fortified sample chromatograms contained only the analyte of interest, and peaks were symmetrical and well defined. Metabolites were expressed in parent equivalents.

The field residue samples were stored frozen a maximum of 723 days (24 months) from harvest to extraction (Table B.7.6.1.1-5A). Residues were determined within 6 days of extraction.

Freezer storage stability data were generated concurrently with the blueberry field trials (Table B.7.6.1.1-5B). Blueberry samples were fortified with 0.1 ppm sulfoxaflor, X11719474, and X11721061 and stored frozen for 756 days. Freezer storage stability recoveries (corrected for concurrent recovery) were within the acceptable 70% to 120% range for all analytes. Therefore sulfoxaflor, X11719474, and X11721061 are stable when stored frozen in blueberries for up to ~25 months, which is longer than trial samples were stored frozen. There are no concerns regarding residue stability in the current study.

| Table B.7.6.1.1-5A. Summary of Storage Conditions. | | | |
|---|---|---|---|
| Matrix | Storage Temperature (°C) | Actual Storage Duration[1] (days/months) | Interval of Demonstrated Storage Stability (days/months) |
| Blueberries | <-20 | 723 days (~24 months) | A concurrent freezer storage stability study was conducted. The data showed that sulfoxaflor, X11719474, and X11721061 residues are stable when stored frozen in blueberries for 756 days (~25 months) (Table B.7.6.1.1-5B). Therefore, there are no concerns regarding residue stability in storage in this study. |

[1] From harvest to residue extraction. Residues were determined within 6 days of extraction.

| Table B.7.6.1.1-5B. Concurrent Freezer Storage Stability Study. | | | | | | |
|---|---|---|---|---|---|---|
| Matrix | Analyte | Storage Period (days) | Fortification Level (ppm) | Freezer Storage Recovery (%) [Average Recovery] | Concurrent Recovery (%) | Corrected Freezer Storage Recovery[1] (%) |
| Blueberries | Sulfoxaflor | 756 (~25 months) | 0.1 | 90, 91, 92 [91] | 84 | 108 |
| | X11719474 | | 0.1 | 84, 85, 85 [85] | 82 | 104 |
| | X11721061 | | 0.1 | 131, 132, 132 [132] | 83 | 120 |

[1] Corrected for recoveries <100% using the following: (Average Freezer Storage Recovery/Concurrent Recovery)*100

The results from these trials showed that when harvested 1 day after the last of 3 applications at a seasonal rate of 0.279 lbs a.i./A (313.1 g ai/ha), average combined residues of sulfoxaflor, X11719474, and X11721061 in blueberries ranged from <0.175 ppm to <1.293 ppm (Tables B.7.6.1.1-6 and B.7.6.1.1-7).

In the residue decline trials, mean residue levels decreased from <0.371 ppm to <0.187 ppm between PHIs of 0 and 21 days at trial site NS321, and from <0.378 to <0.218 between PHIs of 0 and 14 days at trial site OR399.

**Table B.7.6.1.1-6. Residue Data from Blueberry Field Trials with Sulfoxaflor.**

| Location: City, State/Province; Year (Trial ID) | Region | Crop/ Variety | Matrix | End-Use Product | Total Rate (lbs a.i./A) [g a.i./ha] | PHI (days) | Residues[1,2] (ppm) | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | Sulfoxa-flor | X11719474 | X11721061 | Total[3] (per-trial average) |
| Langley, BC; 2014 (BC23) | 12 | Blueberry/ Brigita highbush | Berries | Closer SC | 0.275 [307.6] | 1 | 0.184 | <0.01 | <0.01 | <0.204, <0.146 (<0.175) |
| | | | | | | | 0.126 | <0.01 | <0.01 | |
| Alapha, GA; 2014 (GA*187) | 2 | Blueberry/ TH653 | Berries | Closer SC | 0.267 [298.9] | 1 | 0.420 | <0.01 | <0.01 | <0.440, <0.454 (<0.447) |
| | | | | | | | 0.434 | <0.01 | <0.01 | |
| Jonesboro, ME; 2014 (ME242) | 1 | Blueberry/ Lowbush | Berries | Closer SC | 0.271 [303.7] | 1 | 0.306 | 0.0137 | <0.01 | <0.330, <0.575 (<0.452) |
| | | | | | | | 0.543 | 0.0222 | <0.01 | |
| Fennville, MI; 2014 (MI276) | 5/5A | Blueberry/ Rubel | Berries | Closer SC | 0.279 [313.3] | 1 | 1.390 | 0.0190 | <0.01 | <1.419, <1.166 (<1.293) |
| | | | | | | | 1.140 | 0.0160 | <0.01 | |
| Fennville, MI; 2014 (MI277) | 5/5A | Blueberry/ Bluecrop | Berries | Closer SC | 0.269 [302.4] | 1 | 0.751 | 0.0102 | <0.01 | <0.771, <0.937 (<0.854) |
| | | | | | | | 0.914 | 0.0128 | <0.01 | |
| Fennville, MI; 2014 (MI278) | 5/5A | Blueberry/ Jersey | Berries | Closer SC | 0.270 [302.4] | 1 | 0.642 | <0.01 | <0.01 | <0.662, <0.603 (<0.633) |
| | | | | | | | 0.583 | <0.01 | <0.01 | |
| Cream Ridge, NJ; 2014 (NJ306) | 2 | Blueberry/ Bluecrop | Berries | Closer SC | 0.270 [302.7] | 1 | 0.365 | 0.0148 | <0.01 | <0.39, <0.446 (<0.418) |
| | | | | | | | 0.419 | 0.0173 | <0.01 | |
| Chatsworth, NJ; 2014 (NJ307) | 2 | Blueberry/ Duke | Berries | Closer SC | 0.276 [309.7] | 1 | 0.171 | <0.01 | <0.01 | <0.191, <0.188 (<0.19) |
| | | | | | | | 0.168 | <0.01 | <0.01 | |
| Sheffield Mills, NS; 2014 (NS321) | 1A | Blueberry/ Wild clone lowbush | Berries | Closer SC | 0.274 [307.1] | 0 | 0.346 | <0.01 | <0.01 | <0.366, <0.376 (<0.371) |
| | | | | | | | 0.356 | <0.01 | <0.01 | |
| | | | | | | 1 | 0.312 | <0.01 | <0.01 | <0.332, <0.303 (<0.318) |
| | | | | | | | 0.283 | <0.01 | <0.01 | |
| | | | | | | 8 | 0.203 | <0.01 | <0.01 | <0.223, <0.210 (<0.217) |
| | | | | | | | 0.190 | <0.01 | <0.01 | |
| | | | | | | 14 | 0.168 | <0.01 | <0.01 | <0.188, <0.207 (<0.198) |
| | | | | | | | 0.187 | <0.01 | <0.01 | |
| | | | | | | 21 | 0.163 | <0.01 | <0.01 | <0.183, <0.190 (<0.187) |
| | | | | | | | 0.170 | <0.01 | <0.01 | |
| Milford, NS; 2014 (NS322) | 1A | Blueberry/ Wild clone lowbush | Berries | Closer SC | 0.270 [302.9] | 1 | 0.374 | <0.01 | <0.01 | <0.394, <0.419 (<0.407) |
| | | | | | | | 0.399 | <0.01 | <0.01 | |
| Debert, NS; 2014 (NS323) | 1A | Blueberry/ Wild clone lowbush | Berries | Closer SC | 0.269 [302.2] | 1 | 0.271 | <0.01 | <0.01 | <0.291, <0.285 (<0.288) |
| | | | | | | | 0.265 | <0.01 | <0.01 | |
| Aurora, OR; 2014 (OR399) | 12 | Blueberry/ Bluecrop | Berries | Closer SC | 0.268 [301.0] | 0 | 0.301 | <0.01 | <0.01 | <0.321, <0.434 (<0.378) |
| | | | | | | | 0.413 | 0.0111 | <0.01 | |
| | | | | | | 1 | 0.292 | <0.01 | <0.01 | <0.312, <0.347 (<0.33) |
| | | | | | | | 0.327 | <0.01 | <0.01 | |
| | | | | | | 7 | 0.252 | <0.01 | <0.01 | <0.272, <0.231 (<0.252) |
| | | | | | | | 0.211 | <0.01 | <0.01 | |
| | | | | | | 14 | 0.226 | 0.0117 | <0.01 | <0.248, <0.188 (<0.218) |
| | | | | | | | 0.168 | <0.01 | <0.01 | |

[1] Expressed as parent equivalents. To express the metabolite residues as parent equivalents, residues of each metabolite were multiplied by the ratio of the molecular weights of sulfoxaflor and the respective metabolite. Therefore residues of X11719474 were multiplied by 277.27/295.29,

and residues of X11721061 were multiplied by 277.27/191.15.
[2] Values < LOQ are assumed to be at the LOQ (0.01 ppm).
[3] Total = Sulfoxaflor + X11719474 + X11721061.

**Table B.7.6.1.1-7. Summary of Residues from Blueberry Field Trials with Sulfoxaflor.**

| Crop Matrix | Analyte | Total Application Rate (lbs a.i./A) [g a.i./ha] | PHI (days) | n | Residues[1] (ppm) | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Max.[2] | LAFT[3] | HAFT[3] | Median[3] | Mean[3] | SD[3] |
| Blueberries | Sulfoxaflor | 0.267-0.279 [298.9-313.1] | 1 | 12 | 1.390 | 0.155 | 1.269 | 0.389 | 0.462 | 0.315 |
| | X11719474 | 0.267-0.279 [298.9-313.1] | 1 | 12 | 0.0222 | <0.01 | 0.0180 | <0.01 | <0.0119 | 0.003 |
| | X11721061 | 0.267-0.279 [298.9-313.1] | 1 | 12 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01 | -- |
| | Total[4] | 0.267-0.279 [298.9-313.1] | 1 | 12 | <1.419 | <0.175 | <1.293 | <0.413 | <0.484 | 0.317 |

n = number of independent field trials, LAFT = lowest average field trial, HAFT = highest average field trial, SD = standard deviation

[1] Expressed as parent equivalents.
[2] Values based on total number of samples.
[3] Values based on per-trial averages.
[4] Total = Sulfoxaflor + X11719474 + X11721061.

Note 1: For computation of the LAFT, HAFT, median, mean, and standard deviation, values < LOQ are assumed to be at the LOQ (0.01 ppm).

## III. CONCLUSIONS

The blueberry field trials are considered scientifically acceptable. The results of the study showed that following a total application of 0.279 lbs a.i./A (313.1 g a.i./ha) in blueberry samples collected at PHIs of 1 day, average combined residues of sulfoxaflor, X11719474, and X11721061 ranged from <0.175 ppm to <1.293 ppm. A decline study indicates that the level of residues in blueberries decreases with time. Adequate storage stability data are available to support sample storage durations and conditions.

## REFERENCE

PMRA # 1941241. MRID # 47832031. Rodrigues Junior, A. (2010) “Enforcement Method for the Determination of Sulfoxaflor (XDE-208) and its Main Metabolites in Agricultural Commodities using Offline Solid-Phase Extraction and Liquid Chromatography with Tandem Mass Spectrometry Detection.” Laboratory Study ID: 091116. Unpublished study prepared by Dow AgroSciences Ind. Ltda, Mogi Mirim, SP. 93 pages.